



750 -101
130-105

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of: George G. Wai

Serial No.: 08/689,193 Examiner: Unknown
Filed: 08/05/96 Docket No.: 5404
Title: PITCH CONTROL COMPOSITION

360 TL 10/07/96 09682193
1 101 130.00 CK
750.00 C

RESPONSE TO MISSING PARTS NOTICE

Honorable Commissioner of Patents and Trademarks
Washington, D.C. 20231

Sir:

This is in response to the "Missing Parts Notice" mailed on August 14, 1996.

The Missing Parts Notice stated that the statutory basic filing fee of \$750.00 and the Declaration were missing. It stated that a surcharge of \$130.00 was also due.

The Declaration of the inventor, George K. Wai is enclosed. Attached is a check in the amount of \$880.00 to cover the costs of fees. Please direct any excess funds to Deposit Account No. 02-2530 or likewise charge any deficiencies thereto. A duplicate copy of this transmittal form is enclosed.

A copy of the "Missing Parts Notice" is also enclosed.

Respectfully submitted,

A handwritten signature in black ink that appears to read "David L. Hedden".

David L. Hedden, Attorney
Registration No. 29,388

Phone: (614) 790-4265
Ashland Chemical Company
P.O. Box 2219
Columbus, Ohio 43216

360 TL 10/07/96 09682193
1 101 130.00 CK



-2-

CERTIFICATE OF MAILING

I hereby certify that this Response to Missing Parts Notice is being deposited with the United States Postal Service on September 24, 1996 with sufficient postage as first class mail in an envelope addressed to the Commissioner of Patents and Trademarks, Washington, D.C. 20231.

A handwritten signature in black ink, appearing to read "Janelle Beandy", is written over a single horizontal line.

PITCH CONTROL COMPOSITION

93 AUG -5 AM 8:44

TECHNICAL FIELD OF THE INVENTION

This invention relates to a liquid composition for the control
5 of pitch deposition in pulp and paper making operations comprising
an aqueous solution of (1) a derivatized cationic guar polymer, and
(2) an alkali metal isobutylene/maleic anhydride copolymer. The
invention also relates to a process for inhibiting stickies
deposition on paper making equipment.

10

BACKGROUND OF THE INVENTION

In a broad sense, "pitch" is any "sticky" substance found in
the pulp and papermaking process. These substances create quality
and efficiency problems in the paper making process when they
15 deposit on paper making equipment. More specifically "pitch"
refers to any soluble organic matter originating from the extracts
of wood during pulping which includes fatty acids and esters, resin
acids, and sterols. Pitch may also include process additives such
as defoamers, sizing agents, and coatings, as well as inorganic
20 components like calcium carbonate, silica, clay, magnesium and
titanium. Pitch is released from wood during chemical and
mechanical pulping.

Fine particles of pitch that remain well-dispersed do not
create a deposition problem. However, there is a tendency for the
25 hydrophobic pitch particles to agglomerate at the air-water
interface. These pitch particles form deposits due to their
decreased solubility as they move downstream (through cooler
temperatures and lower pH) and from the increased turbulence of the
process. These pitch particles aggregate to form tacky threads or
30 balls which then deposit on pulp and paper making equipment
including chest walls, screens, paper machine headbox, wires, felts
and dryers. This can also lead to sheet holes and breaks in paper
resulting in increased downtime and/or lower quality paper. Such
pitch deposition can be also be a problem in recycled or secondary
35 fiber processes where synthetic organic polymeric organo-soluble

pitch contaminants called "stickies", such as hot melts, polyethylene films, latexes, pressure sensitive adhesives, and waxes agglomerate and form deposits on papermaking equipment.

Typically used pitch control agents include nonionic surfactants, especially nonylphenol ethoxylates, talc and alum. Nonionic surfactants, which contain both a hydrophobic and hydrophilic segment, decrease the surface tension, provide wetting, detergency and dispersancy. However, nonylphenol ethoxylates have a high foaming tendency which may result in higher defoamer usage, thus depositing more pitch.

Talc is also used to control pitch deposition by adsorbing colloidal pitch onto the talc particles so it is retained in the sheet. The disadvantage of using talc is that it tends to deposit further downstream especially in paper machine felts and cause felt plugging. In acid paper making systems, alum or aluminum sulfate is used to decrease the tackiness of the pitch by cationic fixation to pulp. When using alum, pH control is critical to prevent deposition of alum.

Inorganic dispersants like sodium polyacrylate, and similar compounds, sequester or chelate the inorganic portion of the pitch particle, but leave the organic part free to interfere with low foam tolerant processes as well as increase defoamer usage.

Current technology in pitch or stickies control involves the use of detackifiers, which are hydrophilic polymers, to treat the hydrophobic pitch or stickies. The hydrophilic polymers are adsorbed onto the hydrophobic pitch or stickies particles, thereby converting the hydrophobic pitch or stickies particles to hydrophilic particles. The pitch or stickies particles, which are now hydrophilic, are likely stay with the pulp or paper and less likely to deposit on any kind of machinery, felt or wire surfaces.

Some known detackifiers are polyvinyl alcohol (PVA)¹, methylcellulose², and polyvinylacetate/ethyleneoxide³.

¹ See U.S. Patent 4,871,424.

² See U.S. Patent 4,846,933.

³ See U.S. Patent 5,266,166.

SUMMARY OF THE INVENTION

This invention relates to a liquid composition for the control of pitch deposition in pulp and paper making comprising:

- 5 (1) a derivatized cationic guar polymer, and
- (2) an alkali metal salt of isobutylene/maleic anhydride copolymer.

The invention also relates to a process for inhibiting pitch deposition on paper making equipment. It can be used on hardwood
10 and softwood pulp and papermaking processes for recycled fibers.

The subject compositions not only detackify pitch or stickies particles, but will disperse such particle and prevent them from agglomerating. The ionic balance of the papermaking system is not disturbed, thus limiting detrimental interactions with other
15 process additives. Additionally, the liquid composition does not generate additional foam for the system to handle.

ENABLING DISCLOSURE AND BEST MODE

The derivatized cationic guar polymer used in the pitch inhibiting composition is a powder. Guar gum is a nonionic galactomannan (MW = 250,000 to 2 million) as obtained from the endosperm of the seed of the guar plant. To charge the guar, the nonionic guar is reacted with hydroxypropyl trimonium chloride to a certain degree of substitution which establishes the amount of
25 cationic charge or charge density value in meq/g according to a proprietary process. The resulting cationic guar polymer is a polymer without any significant surface-active properties. Preferred derivatized cationic guars have a charge density of 0.01
meq/g to 3.0 meq/g, preferably 0.01 meq/g to 0.15 meq/g.
30 Particularly preferred as the derivatized cationic guar polymers are hydroxypropyl trimonium chloride, N-HANCE 3000, GALACTOSOL 80H₂C, and JAGUAR 8913, 8914 and 8917.

The isobutylene/maleic anhydride copolymers have an average molecular weight of 5,000 to 100,000, preferably 10,000 to 20,000.
35 They are prepared by the copolymerization of isobutylene and maleic

anhydride monomers according to well known methods in the art. Preferred are the sodium salts of isobutylene/maleic anhydride such as TAMOL 731 supplied by Rohm and Hass Company.

The formulation of the pitch control composition is such that
5 it may contain from 1-20 weight percent cationic guar and 1-20 weight percent isobutylene/maleic anhydride copolymer stabilized in an aqueous solution, said weight percents being based upon the total weight of the aqueous pitch control solution. Preferably the weight ratio of cationic guar to isobutylene/maleic anhydride
10 copolymer is from 6:1 to 1:6, preferably from 5:1 to 1:5, more preferably from 3:1 to 1:3, and most preferably 2.1 to 1.2.

The pitch control composition may be stabilized with an acid, typically up to 0.5 percent by weight, preferably about 0.30 percent by weight (hydrochloric, sulfuric, phosphoric, acetic or
15 nitric) for viscosity modification to impart flow characteristics, where said weight percent is based upon the weight of aqueous pitch control composition.

The invention also relates to a process for inhibiting stickies deposition on paper making equipment, especially when
20 processing recycled paper. The amount of deposit control composition needed to effectively reduce the amount of sticky substances in the paper pulp is from 0.1 lb. to 10 lbs. based upon the weight of the dry fiber. The pitch control composition can be added to any feedpoint in the pulp and papermaking process, for
25 instance the first, second, or third stage washers of the pulp mill, the deckers of the pulpmill, the screens, post bleaching operations, and the paper machine itself.

The pitch control compositions reduce pitch deposition under Kraft brownstock washer/screen room conditions in laboratory
30 simulations at a dosage of 0.10 ppm to 100 ppm as active, preferably at a dosage of 50 ppm, most preferably at a dosage of 15 ppm to 50 ppm and under both acid and alkaline conditions.

Preparation of Synthetic Pitch

The first pitch evaluation test method used involved the addition of a synthetic pitch to a 0.5% consistency of bleached hardwood pulp at 50 C°. The synthetic pitch was a sodium soap of 5 a tall oil fatty acid in ethanol.

The treated samples were prepared by adding aqueous solutions of the polymers listed in the Table I to the pulp slurry an amount of 50 ppm based upon the amount of the sample treated. The polymer solutions were added to the slurry with agitation provided by a 10 propeller-type mixer.

Following the addition of treatment compound, a solution of calcium chloride was added to the slurry to create a colloidal pitch solution. The pH of the slurry dropped from 11.0 to 10.8-10.5 range. Agitation and slurry temperature were maintained for 15 a period of 10 minutes. The mechanical shearing together with the pH drop forced the pitch out of solution and where it was deposited on the sides the steel beaker, and the blades and shaft of the mixer head. After mixing, the beaker and mixer were gently rinsed to remove loose fibers.

20 The amount of deposition was determined by the difference of the initial weights of the beaker and agitator subtracted from the oven-dried final weights. The pitch reduction or inhibition, expressed as a percent, was based on the difference between deposit weight gain on the untreated and treated samples divided 25 by the same untreated sample weight.

The specific pitch control agents tested are set forth in Table I. The Blank did not contain a pitch control agent. Comparison Examples A-E are pitch control agents outside the scope of this invention and were tested for comparison purposes. 30 The pitch control agent used in the Example 1 is within the scope of this invention, and is a blend of a cationic guar and an isobutylene/maleic anhydride copolymer where the weight ratio is 1:1. The results of the tests at a pH of 10.0 to 10.5 are set forth in Table II.

TABLE I
DESCRIPTION OF PITCH CONTROL AGENTS TESTED

5

Pitch Control Agent	Description
Blank	Test with no additive
Comparison A	Nonylphenol ethoxylated with 9 moles of ethylene oxide
Comparison B	Dispersant/surfactant blend
Comparison C	SOKALAN HP 22 (polyoxyethylene/vinyl acetate copolymer) sold by BASF
Comparison D	Derivatized cationic guar
Comparison E	Isobutylene/cationic guar
Blend F/Example 1	Derivatized guar/isobutylene maleic blend (TAMOL 731 sold by Rhom and Haas Company)

TABLE I

**COMPARISON OF BLENDS OF DERIVATIZED CATIONIC GUAR
AND ISOBUTYLENE/MALEIC ANHYDRIDE COPOLYMER
5 WITH OTHER PITCH DISPERSANTS (pH = 10.0-10.5)**

As the results in Table II show, the cationic guar polymer/isobutylene maleic blend of Example 1, having a weight ratio of cationic guar polymer to isobutylene/maleic anhydride copolymer of 1:1, shows the smallest pitch weight gain, or highest percentage pitch reduction, under conditions at a pH 10.0 to 10.5. This indicates the pitch control agent of Example 1 is the best of the 5 products tested, including the patented SOKALAN HP 22 (Blend C). It is also superior to conventional pitch control agents, the foamy surfactant (Blend A) and dispersant/surfactant (Blend B) which were all used successfully in brownstock washer/screen room applications.

Table II

20 **(PITCH DISPERSANT TESTS AT A PULPING pH = 10.0-10.5)**

<u>Treatment</u>	<u>Active Dosage (ppm)</u>	<u>Pitch Deposition (mg)</u>	<u>% Pitch Reduction</u>
Blank	0	109.7	0
Blend A	50	60.4	44.9
Blend B	50	84.8	22.7
Blend C	50	38.1	65.2
Blend D	50	22.7	79.3
Blend E	50	36.1	67.1
Blend F			
Example 1	50	11.2	89.8

Another test was performed to evaluate the efficacy of various treatments for post-bleaching or papermachine applications at a lower pH. The same test procedure was used as that used for the Examples described in Table II, except the 5 final slurry had a pH of 6.5-7.0. The pulp slurry consistency was increased to 1.0%. The results of this test appear in Table III.

10

Table III

(PITCH DISPERSANT TESTS AT A PULPING pH = 6.5-7.0)

<u>Treatment</u>	<u>Active Dosage (ppm)</u>	<u>Pitch Deposition (mg)</u>	<u>% Pitch Reduction</u>
Blank	0	789.7	0
Blend A	10	560.9	29.97
Blend B	10	652.5	11.68
Blend C	10	194.1	73.73
Example 2 (Blend F)	10	93.4	89.31

15

The results in Table III indicate that the cationic guar polymer/isobutylene maleic acid copolymer (Blend F/Example 2) is again better than the SOKALAN HP 22 (Blend B) and the best of all 20 5 tested products in acid to neutral pH, which is a condition normally encountered in papermachine systems.

To further illustrate the effectiveness of the pitch control agents within the scope of this invention, an actual sulfite mill pitch was used as a source of pitch deposit. This test method 25 involved the pulping of a 1.0% consistency bleached hardwood pulp at 45 C°. For one set of this tests, the pH of the slurry was adjusted to 4.0, while in another the slurry pH was at 8.0. A 2" X 3" piece of polyester film, suspended in the slurry, was used as an additional pitch collector.

30 Following treatment addition, the sulfite pitch first dispersed in acetone/hexane solution. This solution was then slowly introduced into the slurry and mixed for an additional 10

RECORDED - INDEXED - SERIALIZED - FILED

minutes while maintaining temperature. At the end of the mixing time, the film, beaker, and mixer were gently rinsed to remove loose fibers. The same percentage pitch reduction calculation was used to determine treatment efficacy. The results of these two sets of tests are in Table IV.

TABLE IV

(TESTS USING SULFITE MILL PITCH)

5

Treatment	SLURRY pH = 4.0			SLURRY pH = 8.0		
	Active Dosage (ppm)	Pitch Deposition (mg)	% Pitch Reduction	Active Dosage (ppm)	Pitch Deposition (mg)	% Pitch Reduction
Blank	0	96	0	0	98.6	0
A	50	75.8	21	50	48.7	50.6
B	50	32	66.7	50	37.6	61.9
C	50	22	77.1	50	30.1	69.5
D	50	28.2	70.6	50	51.4	47.9
EXAMPLE 3 (Blend F)	50	17.5	81.8	50	18.4	81.3

As can be seen from these results, the composition of
 10 cationic guar polymer/isobutylene maleic anhydride copolymer
 Example 3(Blend F), used as a detackifier, is a more effective
 pitch controlling agent than industry accepted products (Blends
 A-D). The results show that Blend F is effective at different pH
 levels which is not observed when the comparison products are
 15 used. These results also confirm the earlier findings, that
 blend E, at equivalent dose, is more effective than the patented
 SOKALAN HP-22.

CLAIMS

We claim:

1. A liquid composition for the control of pitch deposition in
5 pulp and paper making comprising in aqueous solution:
 - (a) a derivatized cationic guar polymer, and
 - (b) a isobutylene/maleic anhydride copolymer having an
10 average molecular weight of from 5,000 to 100,000.
2. The pitch control composition of claim 1 wherein the
isobutylene/maleic anhydride copolymer has an average
molecular weight of from 10,000 to 20,000.
15
3. The pitch control composition of claim 2 wherein the charge
of the cationic guar polymer is derived from hydroxypropyl
trimonium chloride.
20
4. The pitch control composition of claim 3 wherein the charge
density of the derivatized cationic guar polymer is from 0.01
meq/g. to 3.0 meq/g.
25
5. The pitch control composition of claim 4 wherein the
isobutylene/maleic anhydride copolymer has an average
molecular weight of from 10,000 to 20,000.
30
6. A process for controlling the deposition of pitch in a
hardwood or softwood pulp or papermaking process which
comprises applying an effective pitch retarding amount of the
composition of claim 5 to a feedpoint in the pulp or
papermaking process.

7. The process of claim 6 wherein the pitch is pulp mill pitch and the composition of claim 5 wherein the feedpoint is the brown stock washer, screen room, or decker processing areas.

5 8. The process of claim 6 wherein the composition of claim 5 wherein the feedpoint is the post bleaching operation or the paper machine.

9. The process of claim 6 wherein the composition of claim 5 is
10 applied directly to equipment used in pulp and papermaking.

10. The process of claim 6 wherein the composition of claim 5 is applied to the equipment of the pulp and papermaking process and through the shower process water.

15

ABSTRACT

This invention relates to a liquid composition for the control
of pitch deposition in pulp and paper making comprising an aqueous
5 solution of (1) a cationic guar polymer, and (2) isobutylene/maleic
anhydride copolymer. The invention also relates to a process for
inhibiting pitch and stickies deposition in pulp and papermaking
systems.



**COMBINED DECLARATION, POWER OF ATTORNEY
PATENT APPLICATION AND PETITION**

In re application of: George G. Wai

Serial No.: 08/689,193 Examiner: Unknown

Filed: 08/05/96 Docket No.: 5404

Title: PITCH CONTROL COMPOSITION

As a below named inventor, I hereby declare the following:

My residence, post office address and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor of the subject matter which is claimed and for which a patent is sought on the invention entitled:

PITCH CONTROL COMPOSITION
(Title)

the specification of which

is attached hereto.

was filed on _____ as Application Serial No. _____ or Express No. _____, and was amended on _____ (if applicable).

was described and claimed in PCT International Application No. _____, filed on _____, and as amended under PCT Article 19 on _____ (if any).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment referred to above.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, §1.56(a).

I claim foreign priority benefits under Title 35, United States Code, §119 of any foreign application(s) for patent or inventor's certificate listed below and have also identified below any foreign application for patent or inventor's certificate having a filing date before that of the application on which priority is claimed:

App. No.	Country	Filing Date	Priority Claimed Yes <input type="checkbox"/> No <input type="checkbox"/>
----------	---------	-------------	--

I hereby claim the benefit under Title 35, United States Code, §120 of any United States application(s) listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States application in the manner provided by the first paragraph of Title 35, United States Code, §112, I acknowledge the duty to disclose material information as defined in Title 37, Code of Federal Regulations, §1.56(a) which occurred between the filing date of the prior application and the national or PCT international filing date of this application:

(App. No.)	(Filing Date)	(Status)
------------	---------------	----------

I hereby appoint the following attorneys to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

David L Hedden, 29388
Vernon F. Venne, 27014
Mary E. Picken, 30098
Burton A. Amernick, 24852

Address all telephone calls to

David L. Hedden at 614-790-4265.

Address all correspondence to

David L. Hedden
ASHLAND CHEMICAL COMPANY
P.O. Box 2219
Columbus, Ohio 43216.

Wherefore I pray that Letters patents be granted to me for the invention or discovery and claimed in the foregoing specification and claims, and I hereby subscribe my name to the foregoing specification and claims, declaration, power of attorney, and this petition.

I hereby declare that all statements made herein of my knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of

Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

FIRST OR SOLE INVENTOR

Full name of sole or first inventor: George K. Wai

Inventor's signature:

George K. Wai

Date:

Sept. 10, 1996

Residence:

18 Laurelwood Drive
Mountain Lakes, NJ 07046

Citizenship:

Canada

Post Office Address:

(Same as residence)